



CASE 1908

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3/18/3

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

IN RE APPLICATION OF

SHI, ET AL.

S.N. 09/817,419

FILED: 26 MARCH 2001

FOR: CEREAL GRAINS WITH HIGH  
TOTAL DIETARY FIBER AND/OR  
RESISTANT STARCH CONTENT  
AND PREPARATION THEREOF

Group Art Unit: 1761

Examiner: LIEN TRAN

Commissioner of Patents and Trademarks  
Washington, D.C. 20231

DECLARATION UNDER RULE 132 (37 C.F.R. §1.132)

Sir:

I, Dr. Yong-Cheng Shi, currently residing at 133 Terhune Lane, Hillsborough, NJ 08844, USA, do solemnly and sincerely declare as follows:

1. I am currently employed as a Research Manager at National Starch and Chemical Company where I supervise research pertaining to resistant starch.
2. I am a graduate of Zhejiang University (Hangzhou, China) where I was awarded a Bachelor of Science in Chemical Engineering in 1984. I am also a graduate of Kansas State University (Manhattan, Kansas) where I was awarded a Master of Science degree in Grain Science in 1989 and a Doctorate of Philosophy in Cereal Chemistry in 1993.
3. I worked as a graduate research assistant at Kansas State University in the department of Grain Science and Industry from 1987 to 1993 during which time I conducted research on starch structure, properties and modifications. I further conducted post-doctoral work at Kansas State University from 1993 to 1994 on rice and corn grits composition and properties.

4. I taught at Jiangsu Agricultural College in the Department of Food Science from 1984 to 1987.
5. I was awarded:
  - a) the Ming-Long/Paul Seib Graduate Achievement Award at Kansas State University in 1993; and
  - b) the AACC Carbohydrate Division Student Award in 1991.
6. I was a member of:
  - a) the Alpha Mu Grain Science Honorary Society at Kansas State University;
  - b) the American Association of Cereal Chemists, Carbohydrate Division; and
  - c) the Institute of Food Technologist, Carbohydrate Division.
7. I have eight granted US patents and numerous corresponding patents throughout the world pertaining to starch, including two U.S. patents relating to resistant starch with an increased total dietary fiber (TDF) content. I further have ten publications and have made nine presentations pertaining to starch, two publications and two presentations relating to resistant starch and total dietary fiber content thereof.
8. Since 1994 I have worked at National Starch and Chemical Company. My research at National Starch has been significantly related to resistant starch and dietary fiber.
9. I am qualified as an expert in the field of resistant starch and dietary fiber in light of the above education and research.
10. I am one of the inventors on whose behalf this application was filed in the United States Patent and Trademark Office and am familiar with the issues raised in this case.
11. The experiments below were conducted under my supervision and guidance to demonstrate that the grains produced in the example of U.S. Patent No. 5,972,413 issued to Whitney, et al. (Whitney) are gelatinized and no longer birefringent.

## Experimental

The example of Whitney was closely followed with the exception that the cooking apparatus differed as the Whitney apparatus, to the best of my knowledge, is not commercially available. Instead a batch process was used in which 100 grams of wheat berries (Arrowhead Mills, 12.1% moisture) were mixed with 60 grams warm water (70°C) for five minutes which raised the moisture content to 28.9%. Then, 100 grams hot water (98°C) were added and the mixture was kept at a constant temperature for twenty minutes. The final moisture of the cooked grain was 48.8%. The cooked starch was examined by microscopy and using DSC. The same tests were performed on the heat-treated corn starch of the present application (27.5% moisture, 100°C, 16 hours) and on that of the uncooked wheat grains for comparison.

### *Microscopy*

The starch inside the cooked grain and uncooked grain were taken out carefully and dispersed in water for microscopic examination, including under polarized light.

### *DSC*

About 10 mg anhydrous grain was weighed in the high pressure stainless steel pan. Water, at a ratio of 3:1 water:grain, was then injected into the pan. The sample was scanned twice in the DSC from 10°C to 160°C at a heating rate of 10°C/min. Enthalpy ( $\Delta H$ ) of the gelatinization of the cereal grain is calculated by integration of the area under the endothermic peak on the first DSC scan curve. Duplicates were run for each sample and average values are reported.

## Results

### *Microscopy*

Figure 1 shows the starch of the cooked wheat grains.

Figure 2 shows the starch of the uncooked wheat grains.

Figure 3 shows the starch of the heat-treated grains of the present invention.

As can be seen from the micrographs, the starch of the cooked wheat grains (according to the Example of Whitney) are substantially fully cooked and no longer are birefringent under polarized light in contrast to the starch of the uncooked wheat grain and that of the heat-treated corn of the present invention.

#### DSC

| Sample                            | Onset Temp (°C) | Peak Temp (°C) | End Temp (°C) | Delta H (J/g) |
|-----------------------------------|-----------------|----------------|---------------|---------------|
| Uncooked wheat                    | 58.1            | 65.6           | 74.9          | 6.3           |
| Whitney (cooked wheat)            | N/A             | N/A            | N/A           | N/A           |
| Present Application (cooked corn) | 91.0            | 106.7          | 137.1         | 18.45         |

As can be seen from the above data, the cooked wheat of Whitney is already gelatinized and thus no endothermic event is observed from the DSC data. In contrast, the uncooked wheat and the heat-treated corn of the present invention clearly show endothermic peaks as indicated by the DSC data.

#### Conclusions

Wheat grain cooked using the Example of Whitney results in starch which has been gelatinized and is no longer birefringent as claimed in the present invention. The apparatus of Whitney would only tend to more quickly gelatinize the starch of the wheat grain as it would allow better heat circulation and add shear to the process. Further, longer cooking times would not have changed the result as gelatinization and loss of birefringence and granular structure are irreversible processes. Thus, the cooked grain and process of Whitney would not anticipate the present invention.

I further declare that all statements made herein of my own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by a fine or imprisonment or both under 1001 of Title 18 of the United States Code and such willful false statements may jeopardize the validity of the application or any patent issuing thereon.

Signed at Bridgewater, NJ, this March 5, 2003.  
location date

Yong Cheng Shi  
Dr. Yong-Cheng Shi